

NO DRAWINGS

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(54) METHOD OF MANUFACTURE OF AN ACID COMPOUND WITH  
 CONTROLLED RELEASE

(71) We, NESTLE S.A., of La Tour-de-Peilz, Switzerland, a Swiss company, do hereby declare the invention, for which we pray that patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a method of manufacture of an acid compound with controlled release. It relates also to an acid compound with controlled release.

Food in powder form, particularly milk products in powder form, can be kept only a few months. Moreover, the reconstituted product is not always up to the original. In particular, the texture of yoghurt made from spray-dried yoghurt is not satisfactory. In principle, it would be possible to improve its consistency by the addition of thickening materials like gums, but generally, the use of such means for the applications considered is prohibited by the food and milk regulations. Simply adding an acid to a partially acidified milk powder does not give the required consistency either. It was found that to obtain a satisfactory texture with a yoghurt made of partially acidified milk powder for example, it is necessary to complete the acidification very progressively, preferably within half an hour to two hours approximately.

There is therefore a need for an acid compound with controlled release.

It is known, on the other hand, to encapsulate in gums or proteins aromatic compounds for example, which are released by disintegration of the envelope. In its present state, this technique hardly meets the abovementioned need. The gums and proteins being water-soluble, their disintegration is too fast during reconstitution with water. Moreover, addition of gums in milk products is generally not permitted.

The method according to the invention fulfills the abovementioned need while avoiding the inconvenience of known processes.

The present invention provides a method

for the manufacture of an acid compound with controlled release in which an edible acid, solid at ambient temperature or mixed with a solid carrier, is coated with an edible fat solid at ambient temperature and containing an emulsifier. 50

The invention also provides an acid compound with controlled release comprising an edible acid coated with an edible fat solid at ambient temperature and containing an emulsifier. 55

In a preferred embodiment, the acid is citric acid, lactic acid or an acid fruit concentrate. If the acid is in solid form, it should preferably be finely ground in order that the particle size is between 10 and 100 micrometres. If it is in liquid form, it is necessary to mix it before grinding with a solid carrier such as glucose, sucrose or protein. 60

An edible colouring matter and/or aroma can be added to the acid. 65

The melting point of the fat, the composition and the percentage of emulsifier must be chosen in order to obtain the required releasing time for the acid, preferably half an hour to two hours with the liquid normally used for reconstitution, water at ambient temperature for example. 70

The melting point of the fat should preferably be 45 to 60°C, more preferably 50 to 52°C. In some cases it may be advantageous to add to the fat not only an emulsifier, but also an antioxidant such as tocopherol, butylhydroxyanisole or butylhydroxytoluene. 75

Any edible fat solid at ambient temperature is suitable in principle. With yoghurt in powder form, hydrogenated butter oil will be preferred; but hydrogenated cocoa butter or hydrogenated edible oils such as soyabean, sunflower or groundnut oils can be used also. 80

An emulsifier, 0.1 to 10% soyabean lecithin or glyceryl monostearate or 1 to 20% glyceryl polyricinoleate are preferably used. 85

The acid makes 50 to 80% of the total weight of the compound, the balance (20— 90

50%) comprising the edible fat, the emulsifier and as the case may be, antioxidants, aromas and colouring matter.

- 5 Coating the acid with fat can be done for example in a rotating bowl granulator, at a temperature such that the fat is liquid (45 to 60°C) during 15 to 30 minutes. The coated acid is then left to rest at ambient temperature for some 24 hours. The compound is then cooled rapidly to crystallise the fat, for example by dipping in liquid nitrogen. The cooled granules are finally selected with a sieve, their size being preferably between 50 and 250 micrometres.

#### 15 EXAMPLE 1

- 20 300 g of citric acid are finely ground in order that the dimensions of the particles are comprised between 20 and 50 micrometres. 250 g of hydrogenated butter oil (Wiley melting point 50–52°C) are melted and mixed with 50 g of glyceryl polyricinoleate.

- 25 Maintaining the temperature of the mixture at 55 to 60°C, the 300 g of citric acid are coated with the 300 g of fatty mixture in a rotating bowl during 15 to 30 minutes. The granules are left to rest during 24 hours at ambient temperature, then dipped into liquid nitrogen where they are maintained for approximately 5 minutes at –196°C. Finally, the cold granules are passed through a nylon sieve with 50 to 60 micrometre apertures.

- 35 The addition of approximately 4% in weight of the granules obtained to a milk powder partially acidified with *Lactobacillus bugarius* and *Streptococcus thermophilus* gives, on reconstitution with water, the texture of a gel which is usual and desirable for this kind of product.

- 40 With a percentage of solid matter of 19%, the pH of the yoghurt is 5.6 just after reconstitution and 4.6 two hours later.

#### 45 EXAMPLE 2

- 50 Granules comprising 22.5% liquid lactic acid and 2.5% water mixed with 25% glucose, 46% hydrogenated butter oil (Wiley melting point 50–52°C) and 4% glyceryl polyricinoleate are prepared as in Example 1. These granules are added to a whole milk powder (not acidified) containing 14% milk fat and 82% milk solids non-fat in the proportion of 19.0 g of granules for 100 g of milk powder. The mixture is then diluted in a proportion of 18.4 g of powder for 81.6 g of water at 35 to 40°C. After about one hour, the suspension has a gelled texture.

#### 60 EXAMPLE 3

- 65 The same granules as in Example 2 are added to a non-acidified whole milk powder containing 17% milk fat, 46% milk solids

non-fat and 33.5% of glucides, in a proportion of 7.5 g of granules for 100 g of milk powder. The whole is diluted in a proportion of 20.6 g of powder to 79.4 g of water at 35 to 40°C. After one hour approximately the suspension has a partially gelled structure.

70 The use of an acid compound with controlled release in the production of milk products such as is described above in the examples is described and claimed in our co-pending Application No. 4038/72 (Serial No. 1,330,793).

#### 80 WHAT WE CLAIM IS:—

1. A method for the manufacture of an acid compound with controlled release in which an edible acid solid at ambient temperature or mixed with a solid carrier, is coated with an edible fat solid at ambient temperature and containing an emulsifier.

2. A method according to claim 1 in which the fat is an edible hydrogenated fat.

3. A method according to claim 1 in which the fat is hydrogenated butter oil.

4. A method according to any of claims 1 to 3 in which the acid is citric acid.

5. A method according to any of claims 1 to 3 in which the acid is lactic acid.

6. A method according to any of claims 1 to 3 in which the acid is an acid fruit concentrate.

7. A method according to any of claims 1 to 3 in which an acid in liquid form is mixed with a solid carrier.

8. A method according to any of claims 1 to 3 in which the acid solid at ambient temperature is ground, the particle size being between 10 and 100 micrometres.

9. A method according to any of claims 1 to 8 in which an aroma is added to the acid.

10. A method according to any of claims 1 to 9 in which an antioxidant is added to the fat.

11. A method for the manufacture of an acid compound of controlled release substantially as hereinbefore described with reference to any of the Examples.

12. An acid compound of controlled release when prepared by a process as claimed in any of claims 1 to 11.

13. An acid compound with controlled release comprising an edible acid coated with an edible fat solid at ambient temperature and containing an emulsifier.

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